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SEPARATION OF GRAPHITE, BIOTITE AND MICACEOUS MINERALS FROM A NOVA SCOTIA GARNET SCHIST

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August 1988

Project: 30.21.02
Beneficiation of Industrial Minerals

BMP

MINERAL SCIENCES LABORATORIES
DIVISION REPORT MSL 88-100 (IR)

MICROMEDIA

SEPARATION OF GRAPHITE, BIOTITE AND MICACEOUS MINERALS FROM A NOVA SCOTIA GARNET SCHIST

by

P.R.A. Andrews*

ABSTRACT

A preliminary evaluation was conducted to concentrate garnet from a graphitic, micaceous garnet schist from Chegoggin Point, Nova Scotia. Graphite and illite were successfully removed by a combination of scrubbing and washing. The best scrubbing time was determined as 20 min; scrubbing times in excess of 20 min resulted in abraded garnet surfaces.

Gravity concentration employing laboratory shaking tables concentrated garnet in the -840 +420 μm fraction to 70-75% garnet; in the -420 +150 μm fraction to 80-85% garnet and in the -150 μm fraction to 95% garnet. Although garnet was effectively liberated in the -2.4 mm +840 μm fraction, concentration by tabling was not possible due to a combined effect of similarity in shape and specific gravity between garnet and chlorite, which was more critical at coarser sizes than finer sizes.

Garnet in the -840 +420 μm fraction was further concentrated by reverse flotation of chlorite to 85% garnet. Chlorite was floated at pH 8.0, after prior grinding and desliming at 45 μm to remove illite, using a tertiary amine collector Ethomeen T 25. A chlorite product analyzing 70% chlorite was obtained with 384 g/t of collector. At this pH garnet was unresponsive to Ethomeen T 25.

Keywords: garnet, graphite, chlorite, gravity concentration, flotation

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SÉPARATION DU GRAPHITE, DE LA BIOTITE ET DES MINÉRAUX MICACÉS D'UN SCHISTE À GRENAT DE LA NOUVELLE-ÉCOSSE

par

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RÉSUMÉ

Une première évaluation a été effectuée pour concentrer le grenat provenant d'un schiste à grenat graphiteux et micacé de Chegoggin Point en Nouvelle-Écosse. Le graphite et l'illite ont été séparés avec succès par épuration et lavage combinés. Le meilleur temps pour l'épuration a été déterminé à 20 minutes; des temps d'épuration plus longs ont donné des surfaces abrasées de grenat.

La concentration par gravité au moyen de tables à secousses de laboratoire a donné dans la fraction -840 +420 µm, entre 70 % et 75 % de grenat; dans la fraction -420 +150 µm de 81 % à 85 %, et dans la fraction -150µm, 95 %. Bien que le grenat soit libéré dans la fraction -2,4 mm +840 µm, sa concentration par tables à secousses n'a pas été possible du fait que le grenat et la chlorite étaient de forme et de densité semblables, similitude qui était plus prononcée pour les grains grossiers que pour les grains fins.

On a poussé à 85 % la concentration du grenat présent dans la fraction -840 +420 µm par flottation inverse de la chlorite. La chlorite était séparée par flotation à pH 8, après broyage et déschlammage à 45 µm pour éliminer l'illite, en présence du collecteur Ethomeen T 25, qui est une amine tertiaire. On a obtenu un produit chloriteux renfermant 70 % de chlorite avec 384 g de collecteur par tonne. À ce pH, le grenat était réfractaire à l'Ethomeen T 25.

Mots clés : grenat, graphite, chlorite, concentration par gravité, flottation

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INTRODUCTION

In October 1987, R. MacDonald of the Nova Scotia Department of Mines, Stellerton, Nova Scotia, discussed with the Industrial Minerals Section the possibility of concentrating garnet from a deposit occurring at Chegoggin Point, Nova Scotia. Subsequently, a 20 kg sample of garnet schist was received in November 1987.

The presence of garnetiferous schist at Chegoggin Point has been known for many years. The deposit was originally reported by Faribault (1919) and preliminary concentration tests conducted by Flynn (1923) and again by Cameron (1952). Results of testwork indicated that concentration of garnet using magnetic and gravity concentration techniques was only partially successful. Magnetic separation produced 60% garnet concentrates at sizes below 600 μm; chlorite could not be effectively separated. Similarly, with tabling at sizes below 800 μm no definite division between concentrate, middling and tailing was possible due to the presence of chlorite.

Before 1987 very little commercial interest was shown in the garnet deposits, although the white quartzite, which overlies the garnet schist, was mined by Dominion Steel and Coal Corporation.

GEOLOGY

The strata at Chegoggin Point cuts the coast line obliquely, the regional strike being N35° to 40°E, magnetic, and the dip approximately 80°E. As exposed along the beach, the rocks are highly metamorphosed to various crystalline schists and slates of varying composition, being micaceous, hornblendic, pyritic or garnetiferous. Exposures of relatively pure hornblende were noted and in places the weaker mica schists curve around the hornblende masses. Several quartz veins were noted, which cut the strata at a very low angle. The quartz veins, as exposed, averaged 200 to 250 mm wide, but were discontinuous over short distances.

Various beds were identified; some appeared to be predominately mica schists containing a few small garnets, while other schists were rich in garnets, the estimated bed thickness being approximately 3 m.

SAMPLE DESCRIPTION

Approximately 20 kg of a garnetiferous graphite micaceous schist, consisting of several large 200 mm ore specimens were received at the Industrial Minerals Laboratory in November 1987. The garnet crystals were subhedral to euhedral in shape ranging in size from 1 mm to 6 mm; the majority being 3 to 4 mm in size, and the entire mineral surfaces were coated with a film of graphite. Other impurities included minor amounts of quartz, and some iron staining on the micaceous minerals which indicated the presence of non-sulphides. The garnet content was estimated to be 50-60% and garnet grains appeared to have a hackly-chisel fracture when crushed with no visible magnetite inclusions.

PROCESSING

Size Reduction

The 20 kg sample was crushed to pass 6 mm and examined visually to assess coarse liberation of garnet. Since there was virtually no coarse liberation, size reduction was continued to 2.4 mm.

Head Sample

A representative 200 g head sample was screened from 2.4 mm to 150 µm and each fraction examined by binocular microscopy to determine mineral content and degree of liberation with decreasing size; results are reported in Table 1.

Secondary Crushing and Sizing

The remainder of the 20 kg sample was further size reduced by roll crushing to pass 840 µm, the garnet liberation size, and sized at -840 +420 µm, -420 +150 µm and -150 µm.

Scrubbing

Visual examination of the three size fractions suggested that processing of the intermediate -420 +150 µm fraction would benefit from a preliminary attrition scrubbing to remove graphitic and chloritic slime. A

1 kg portion was scrubbed at 85% solids in a laboratory Wemco attrition scrubber for 30 min and wet screened at 150 μm to remove slime-forming minerals. Visual assessment indicated that the garnet surfaces were free of graphite, but the surfaces had become abraded and rounded by attrition.

Three scrubbing tests were conducted for 10 min, 20 min and 30 min scrubbing times to determine the optimum time which would least abrade garnet surfaces, but still be sufficient to liberate graphite and micaceous mineral slimes. Visual assessment indicated that scrubbing time should not exceed 20 min.

Gravity Concentration

Portions of -2.4 mm +840 μm, -840 +420 μm and -420 +150 μm size fractions were passed over a Wilfley shaking table, and the -150 μm fraction over a Wilfley slime table to ascertain how efficiently garnet would be concentrated by gravity concentration techniques. The -420 +150 μm fraction was the only size fraction that was scrubbed prior to tabling to remove graphite and micaceous mineral slimes. Visual observations of all products from tabling are reported in Table 2. Product balances for the -420 +150 μm and -150 μm fraction tests, based on X-ray diffraction data, are reported in Table 3.

Graphite Flotation

It was observed during scrubbing tests that liberated graphite formed a film on the surface of the attrition-product mix. A 1 kg sample of -420 +150 μm material was scrubbed for 20 min with NaOH, fuel oil and MIBC (methyl iso-butyl carbinol) frother. After scrubbing a standard graphite float was conducted in a laboratory Denver flotation cell and the graphite concentrate analyzed for carbon.

Micaceous Minerals Flotation

The products from the -840 +420 μm gravity concentration test were re-combined and crushed to pass 600 μm, and riffled into 500 g lots for flotation. Each 500 g lot was wet-ground at 50% solids in a porcelain jar mill with burundum grinding media, and transferred to the laboratory Denver flotation cell. Flotation of micaceous minerals from garnet was conducted

using two flotation procedures. A primary amine, Armac T, tallow amine acetate was employed at pH 8, and a tertiary amine, Ethomeen T 25, polyoxyethylene tallow amine between pH 7.0 and 9.4. Selected micaceous products and garnet concentrates were examined by binocular microscope and analyzed by X-ray diffraction. Flotation testwork product balances are reported in Table 4, and flotation parameter and reagent concentration data in Table 5.

Analysis

X-ray diffraction analysis is a semi-quantitative technique and accuracy is interpreted as ±10%, although analyses are reported to the nearest 5%.

RESULTS

Although garnet was approximately 90% liberated from chlorite and graphite in the -2.4 mm +840 μm fraction, a separation, using a laboratory shaking table was not possible due to a combined effect of similarity in shape and specific gravity between garnet and chlorite, which was more critical at coarser sizes than finer sizes. Better separation between garnet and chlorite might be obtained by jigging at this coarse size. Concentration of garnet improved with decreasing size fractions from 70-75% garnet at -840 +420 μm; 80 to 85% garnet at -420 +150 μm and 95% garnet at -150 μm. Although the principal gangue minerals were chlorite and graphite, the presence of illite in the -150 μm was a major contaminant mineral in this fraction.

The results of a preliminary graphite flotation test indicated that a rougher concentrate analyzing 7.5% C could be obtained. Cleaning would undoubtedly increase the carbon content but this was not attempted.

Further concentration of garnet in the -840 +420 μm fraction was attempted by flotation. Flotation of garnet using the primary amine collector, Armac T, (Test 1) did not significantly increase the grade of garnet concentrate. Better results were obtained with reverse flotation of chlorite using the tertiary amine collector Ethomeen T 25. In Test 2, after grinding, a desliming stage at 25 μm removed 30.6% of the illite for a loss

of only 6.9% of the garnet. Flotation removed 32.2% of the chlorite, so that while 92.1% of the garnet was recovered, the concentrate (rougher tails product) was only upgraded to 80%. In Test 3, the desliming size was increased to 45 μm at which size 24.4% of the illite and 42.0% of the chlorite were removed. The increase in the rejection of chlorite indicates that chlorite particles are coarser than 25 μm. Flotation rejected a further 36.6% of the chlorite so that the garnet concentrate (rougher tails product) was increased to 85% garnet. The recovery at 85.3% was less than in Test 1 due to the greater loss of garnet, 14.1%, in the slime fraction. In Test 4, the desliming size was further increased to 70 μm to ascertain whether greater weight rejection of slime material would improve flotation. At 70 μm the rejection of chlorite increased to 59.9%, the illite rejection was approximately the same as in Tests 1 and 2, at 31.9%; garnet loss increased to 17.0%. Flotation rejected a further 25.1% chlorite but the grade of garnet concentrate was not increased above that obtained in Test 3. In Test 5, the grind time was increased in an attempt to ascertain whether a finer grind would liberate more gangue and improve the selectivity of the garnet concentrate. The results show that while a 90% garnet concentrate (rougher tails product) was obtained, the recovery was reduced to 51%. The principal reason for the reduced recovery being the loss of 37.1% of the garnet in the initial desliming stage.

PHYSICO-CHEMICAL CONSIDERATIONS

Surface Charge

The zero point of charge of garnet has been variously determined between 4.4 (Cases, 1970) and 5.8 (Fuerstenau & Fuerstenau, 1983). Garnet belongs to the orthosilicate crystal group and is characterized by non-sharing of oxygen atoms within the SiO_4 structural unit. This structure on fracturing produces a polar surface and exposed Fe^{3+} and Al^{3+} become hydrolized; as such H^+ and OH^- would be expected to be potential determining ions. Indirect evidence that Fe^{3+} and Al^{3+} can affect the z.p.c. was demonstrated by comparing the flotation behaviour of acid-treated garnet with untreated garnet; acid-treatment reduces the flotation response.

The zero point of charge of chlorite has not been determined; the z.p.c. of a similar sheet silicate mineral, biotite, is 0.4 (Fuerstenau & Fuerstenau, 1983). The z.p.c. of chlorite would be expected to be similar, but variability is suspected due to the presence of Fe^{3+}, Al^{3+} and Mg^{2+} as replacement cations in the mineral lattice. Abramov, 1965 has made an extensive study of the surface properties of chlorite and concluded that the surface consists both of partly hydrophobic and hydrated portions. The existence of weakly acidic Si-OH and amphoteric Al-OH layers are thought to influence floatability.

Adsorption Mechanisms

Garnet responds to anionic and cationic flotation. When employing oleic acid, evidence of chemisorption and electrostatic adsorption from infra-red spectroscopy studies was demonstrated by Pol'kin, Kolmogorova and Solo'ev, 1971. The garnet spectra showed bands for Fe and Al oleates in addition to bands characteristic of the undissociated acid. Adsorption of dodecylamine by electrokinetic potential measurements indicated chemical adsorption of amine ions on the garnet surface and physical adsorption of undissociated amine molecules in the diffused portion of the electrical double layer (Naifonov, 1967).

Chlorite responds to anionic and cationic flotation; in fact chlorite displays the largest flotation area of the sheet-silicate minerals. Manser, 1975 found that there was no lower pH limit with either oleic acid or dodecylamine and only the anionic collector displayed an upper pH limit of between pH 8 and 10. There is an absence of fundamental adsorption data for chlorite but such positive response to flotation would imply both chemisorption and electrostatic adsorption.

Garnet-Chlorite Separation

The flotation of chlorite at pH 8 using the tertiary amine collector Ethomeen T 25 is most likely to occur in an area of negative surface potential and would indicate electrostatic adsorption. Proof of electrostatic adsorption would be demonstrated by non-flotation at pH values below the z.p.c. when chlorite would be positively charged. At pH 8 garnet is unresponsive to Ethomeen T 25.

CONCLUSIONS

Scrubbing effectively removed graphite from garnet surfaces; washing and desliming removed both graphite and illite. Gravity concentration using laboratory shaking tables concentrated garnet in the -840 +420 μm fraction from 70-75% garnet; in the -420 +150 μm fraction from 80-85% garnet and in the -150 μm fraction to 95% garnet. Although garnet was effectively liberated in the -2.4 mm +840 μm fraction, concentration by laboratory shaking table was not possible due to a combined effect of similarity in shape and specific gravity between garnet and chlorite, which was more critical at coarser sizes than finer sizes. By grinding the -840 +420 μm fraction, flotation further improved the garnet concentration from 70 to 75% garnet, to 85% garnet. Further increase in grade to 90% garnet was possible but only with a reduced recovery.

RECOMMENDATIONS

Further testwork at the laboratory and possibly pilot-plant level should be conducted with a larger sample to establish a more selective beneficiation procedure. If garnet from Chegoggin Point can be beneficiated to the extent required for its various uses then the following recommendations would apply:

1. A market study should be conducted to establish size and variety of markets, competition from substitute materials and product evaluation with specifications for the requirements pertaining to the type and acceptable level of deleterious material.

2. Cost benefit analysis should be conducted to establish cut-off grade.

3. Additional diamond drilling should be undertaken to determine ore grade and extent of the deposit.

ACKNOWLEDGEMENTS

The author would like to thank the following CANMET personnel for their assistance in this testwork: L. Morin for flotation testwork, W. Kelly for gravity concentration and microscopic evaluation, K. Besso for X-ray diffraction data.

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Table 1. Mineralogy of sized fractions

Size fraction μm		Description
	+9.5 mm	Aggregates of euhedral garnet crystals are locked in a chlorite-biotite-graphitic schist; minor quartz present, minor limonitic staining. Graphite coats the unfractured garnet surfaces.
-9.5	+6.7 mm	Similar to +9.5 mm fraction.
-6.7	+3.4 mm	50 to 60% of this fraction euhedral garnet with the crystal surfaces coated with graphite; liberation of garnet from matrix complete.
-3.4	+2.4 mm	Similar to -6.7 +3.4 mm fraction; combined -6.7 +2.4 mm fraction accounts for most of the garnet.
-2.4	+1.2 mm	Garnet crystals are beginning to be fractured; graphite surface coating is still present.
-1.2 mm	+600 μm	Similar to -2.4 + 1.2 mm fraction.
-600	+300 μm	Garnet grains are free of graphite coating; only 15 to 20% of this fraction is garnet.
-300	+150 μm	Garnet grains are clean but only 20 to 30% of the fraction is garnet; biotite/chlorite well delaminated.
	-150 μm	Garnet accounts for 15% and quartz 10% of this fraction; majority is biotite/chlorite.

Table 2. Shaking table gravity concentration testwork

Size fraction	Remarks
-2.4 mm +840 μm	Although garnet was approximately 90% liberated from chlorite and graphite, some garnet faces were still coated by graphite. No visible concentration was observed.
-840 +420 μm	Concentration was estimated at 70 to 75% garnet. The garnet faces were clean and angular displaying the chisel fracture. Garnet content in the tails was estimated to be 5%.
-420 +150 μm	The concentrate and middlings fractions were estimated to contain 80 to 85% garnet. The garnet faces were extremely clean with good angularity. Minor contaminant minerals included quartz with magnetite inclusions, and chlorite. The tails were mainly chlorite with minor garnet and illite. This fraction was the only fraction that was scrubbed to remove illite prior to tabling.
-150 μm	Concentration was estimated at 95% garnet.

Table 3. Product balances for selected gravity concentration testwork

Products	Weight %	Analysis %				Distribution %			
		Garnet	Quartz	Chlorite	Illite	Garnet	Quartz	Chlorite	Illite
-420 +150 µm									
Concentrate	47.3	78	14	8	-	64.9	80.2	13.0	-
Middling	20.5	85	8	7	-	30.6	19.8	4.9	-
Concn + Midd.	67.8	80	12	8	-	95.5	100.0	17.9	-
Tailings	32.2	8	-	74	19	4.5	-	82.1	100.0
Calculated head	100.0	57	8	29	6	100.0	100.0	100.0	100.0
-150 µm									
Concentrate	36.3	96	-	4	-	63.9	-	6.3	-
Middling	27.4	56	-	27	17	28.1	-	31.9	45.9
Tailings 1	36.3	12	-	40	31	8.0	-	61.8	54.1
Calculated head	100.0	55	-	23	22	100.0	-	100.0	100.0

Table 4. Flotation testwork product balances

Test No.	Products	Weight %	Analysis %			Distribution %		
			Garnet	Chlorite	Illite	Garnet	Chlorite	Illite
1	Rougher float	84.0	70	15	15	-	-	-
	Rougher tails	16.0	-	-	-	-	-	-
	Calculated head	100.0	-	-	-	-	-	-
2	Slime	8.4	60	35	5	6.9	17.5	30.6
	Rougher float	7.2	10	75	15	1.0	32.2	7.9
	Rougher tails	84.4	80	10	10	92.1	50.3	61.5
	Calculated head	100.0	73	17	14	100.0	100.0	100.0
3	Slime	20.0	50	35	15	14.1	42.0	24.4
	Rougher float	8.7	5	70	25	0.6	36.6	17.7
	Rougher tails	71.3	85	5	10	85.3	21.4	57.9
	Calculated head	100.0	71	17	12	100.0	100.0	100.0
4	Slime	28.0	40	45	15	17.0	59.9	31.9
	Rougher float	8.8	10	60	30	1.6	25.1	20.1
	Rougher tails	63.2	85	5	10	81.4	15.0	48.0
	Calculated head	100.0	66	21	13	100.0	100.0	100.0
5	Slime	43.5	60	25	15	37.1	63.8	64.0
	Rougher float	16.7	50	25	25	11.9	24.5	16.5
	Rougher tails	39.8	90	5	5	51.0	11.7	19.5
	Calculated head	100.0	70	17	10	100.0	100.0	100.0

Table 5. Flotation parameter and reagent concentration data

Test No.	Flotation stage	No. of stages	Grind time min	Deslime size µm	Float time min	pH	Collectors		Frother
							Arm. T g/t	Eth T 25 g/t	M.I.B.C. g/t
1	Rougher	3	10	-	8.5	8.2	782.7	-	75.1
2	Rougher	2	10	-25	5	9.5	-	557.4	80.3
3	Rougher	2	10	-45	5.5	8.0	-	384.3	46.1
4	Rougher	4	10	-70	11.5	8.2	-	862.6	135.9
5	Rougher	4	20	-70	11.5	8.3	-	1453.0	171.7